RADIOCHEMICAL ANALYSIS OF VEGETATION REFERENCE MATERIALS CLV-1 and CLV-2

Performed by Chemex Labs Ltd.,
212 Brooksbank Ave.,
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Radiochemical Analyst: Eva Savoy

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Radionuclide Methodology

The radionuclides analyzed as part of this contract include Pb-210, Po-210, Th-230 and Ra-226. The methods used for each radioisotope are outlined below. For those isotopes which were quantified by either gross alpha or gross beta counting techniques following radiochemical separations, the measurements were performed using a Canberra Model 2200 Alpha, Beta Analyzer. This instrument consists of an external proportional counter with an ultra-thin detector window. The counter is completely surrounded by four inches of virgin lead and utilizes ultrahigh purity P-10 counting gas (90% argon - 10% methane).

A. Pb-210:

Because of the weak beta emission from Pb-210, the determination of Pb-210 was carried out indirectly by measurment of its high energy beta-emitting daughter Bi-210.

A mixed carrier solution containing stable lead and bismuth was added to the 5.000 gm samples which were then digested to dryness with a mixture of nitric, hydrofluoric and perchloric acids. If any residue remained, the digestion was repeated to yield a clear digest when taken up in 2M HCL. Lead and bismuth were extracted into chloroform with diethyldithiocarbamate and then back extracted into aqueous solution. The bismuth concentration of this aqueous solution was then determined by

atomic absorption spectrophotometry to determine the recovery of bismuth (typically 95-100%) through the extraction procedure. The bismuth was subsequently precipitated and purified as the oxychloride. The precipitate was filtered through a 0.45u membrane filter in a ring and disc filter assembly, air-dried briefly and then covered with aluminum foil. The aluminum foil serves to screen out weak beta emission from Pb-210 as well as alpha emission from Po-210 which is carried through the procedure.

Samples were counted for 100 minutes and compared to the activities of standard Pb-210 and Bi-210 solutions carried through the same procedure. Certified reference materials were also analyzed with each batch. The standard Pb-210 solution, with which Bi-210 is certified to be in equilibrium, was purchased from Amersham Radiochemical Ltd. and was certified to have a total uncertainty of not greater than 2.3%.

Ref: Canmet Report No. 78-22, G. Smithson, Ed. (1979) pp. 19-22.

B. Po-210:

Samples (5.000 gm) for polonium analysis were digested in Teflon beakers with a mixture of nitric, hydrofluoric and perchloric acids. The mixture was taken to perchloric acid fumes but not to dryness. The digested solutions were diluted, reduced with ascorbic acid then heated for two hours in contact with a silver disc. Under these conditions, polonium spontaneously deposits onto the silver disc which is subsequently counted for its alpha activity. Samples were counted for 100 minutes and compared to the activity of standard Po-210 solutions carried through the same procedure. Certified reference materials were also analyzed with each

batch. The standard Po-210 solution was purchased from Amersham Chemicals and was certified to have a total uncertainty not exceeding 2.3%.

Ref. Canmet Report No. 78-22, G. Smithson, Ed. (1979) pp. 23-28.

C. Th-230:

Th-230 was determined by alpha spectroscopy using an Ortec Model 496 alpha spectrometer with two ruggedized silicon surface barrier detectors. The alpha spectrometer has a spectrum storage capability of 120 channels over a 3MeV range. The multichannel analyzer is controlled directly by a PDP 11/34 computer with 256K bytes of memory, four hard surface discs, a nine-track 1600 bpi tape drive and five different video or typewriter terminals.

Samples (10.000 gm) for Th-230 determination were spiked with beta-emitting Th-234 as a radiotracer, lanthanum chloride as a carrier and barium nitrate as a holdback carrier for radium. This mixture was then ashed at 550 Deg. C and the ash digested with nitric, hydrofluoric and perchloric acids until there was no undissolved residue. Thorium isotopes were then coprecipitated with lanthanum hydroxide, filtered, redissolved and coprecipitated with lanthanum fluoride. The thorium isotopes were then collected on 0.45u filters and alpha-counted for 100 minutes using the instrumentation described above. The samples were also beta-counted for Th-234 to determine the overall yield of the separation. In addition one sample of each type was spiked with Th-230 to verify that counting efficiencies were not compromised by matrix self-absorption.

The activity of samples was compared with the activity of standard Th-230 solutions carried through the same procedure. Certified reference

materials were also analyzed with each batch of samples. The standard Th-230 solution was obtained from Isotope Products Laboratories and had a total uncertainty not exceeding 5.95% on its calibration.

D. Ra-226:

Samples (10.000 gm) for Ra-226 were spiked with Ba-133 as a radiotracer and ashed at 550 Deg. C. The resulting ash was dissolved in a mixture of hot concentrated nitric, perchloric and hydrofluoric acids. Radium was precipitated from solution by coprecipitation with lead sulfate, redissolved and purified by coprecipitation with barium sulfate. The precipitate was collected, gamma counted for Ba-133 to measure the overall yield (90-100%) and then stored for a minimum of one week to allow for the ingrowth of daughters. At the end of this period, samples were counted for 100 minutes using gross alpha counting techniques and compared to the activity of standard Ra-226 solutions carried through the same procedure. The standard Ra-226 solutions were diluted from stock obtained from Amersham Radiochemicals. This solution had been previously calibrated against a U.S. National Bureau of Standards Ra-226 source and has a total uncertainty of not greater than +/-3.9% in its activity.

TABLE OF RESULTS

Vegetation Standard CLV-1

Isotope	Activity (Bq/g)	Uncertainty (Bq/g, 95%C.L.)	Subsample Mass(g)	Date of Analysis
Pb - 210	0.63	0.02	5.000	Mar. 18, 1985
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Pb - 210	0.63	0.02	5.000	Mar. 18, 1985
Pb - 210	0.63	0.02	5.000	Mar. 18, 1985
Po - 210	0.46	0.03	1.000	Mar. 20, 1985
Po - 210	0.45	0.01	5.000	Mar. 15, 1985
Po - 210	0.41	0.03	1.000	Mar. 20, 1985
Po - 210	0.41	0.01	5.000	Mar. 15, 1985
Th - 230	0.32	0.04	2.000	
Th - 230	0.28	0.04	2.000	
Th - 230	0.30	0.04	2.000	
Th - 230	0.29	0.04	2.000	
Ra - 226	0.70	0.01	10.005	
Ra - 226	0.71	0.01	10.012	
Ra - 226	0.68	0.02	2.000	
Ra - 226	0.65	0.03	1.500	

TABLE OF RESULTS

Vegetation Standard CLV-2

Isotope	Activity (Bq/g)	Uncertainty (Bq/g, 95%C.L.)	Subsample Mass(g)	Date of Analysis
Pb - 210	0.066	0.009	5.000	Mar. 19, 1985
Pb - 210	0.065	0.009	5.000	Mar. 19, 1985
Pb - 210	0.059	0.008	5.000	Mar. 19, 1985
Pb - 210	0.067	0.009	5.000	Mar. 19, 1985
Po - 210	0.065	0.006	5.000	Mar. 16, 1985
Po - 210	0.065	0.006	5.000	Mar. 16, 1985
Po - 210	0.059	0.005	5.000	Mar. 16, 1985
Po - 210	0.066	0.006	5.000	Mar. 16, 1985
Th - 230	0.04	0.01	2.000	
Th - 230	0.03	0.01	2.000	
Th - 230	0.03	0.01	2.000	
Th - 230	0.03	0.01	2.000	
Ra - 226	0.014	0.001	10.001	
Ra - 226	0.014	0.001	10.002	
Ra - 226	0.013	0.001	10.001	
Ra - 226	0.014	0.001	10.005	

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Chemex Labs Ltd.
212 Brooksbank Avenue
North Vancouver, B.C. V7J 2C1
Attn: Michael LeGeytt

Date Samples Received: 04-Mar-85 Client P.O.:

SAMPLE CLIENT DESCRIPTION
2065 CLV 1 TEST SAMPLE (FROM EMR CANMET) *VEGETATION*
2066 CLV 2 -65 MESH TEST SAMPLE (FROM EMR CANMET) *VEGETATION*

ANALYTE	UNITS	2065	2066
RADIONUCLIDES			
Pb 210, total	Bq/g	0.75	0.09
Pb 210, total	Bq/g	0.72	0.08
Pb 210, total	Bq/g	0.75	0.08
Pb 210, total	Bq/g	0.72	0.08
Po 210, total	Bq/g	0.35	0.06
Po 210, total	Bq/g	0.38	0.06
Po 210, total	Bq/g	0.36	0.07
Po 210, total	Bq/g	0.32	0.06
Ra 226, total	Bq/g	0.71	0.05
Ra 226, total	Bq/g	0.70	0.06
Ra 226, total	Bq/g	0.73	0.05
Ra 226, total	Bq/g	0.72	0.05
Th 230, total	Bq/g	0.32	0.03
Th 230, total	Bq/g	0.30	0.03
Th 230, total	Bq/g	0.29	0.03
Th 230, total	Bq/g	0.33	0.03

Each sample is prepared and analyzed in quadruplicate.

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Thorium-230

Analyst: Dave Chorney

Date analyzed: March 7, 1985

Tracer: Thorium-234

Sample decomposition: Potassium pyrosulphate fusion

Thorium is separated from prepared sample solutions by coprecipitation with barium sulphate. Barium sulphate is dissolved in a strongly alkaline solution of ethylenediaminetetraacidic acid. Thorium is precipitated as hydroxide with 50 µg cerium carrier. The precipitate is mounted on a 25 mm 0.1 micron membrane filter for alpha spectrometry. Alpha spectrometry is performed using a vacuum chamber mounted surface barrier detector.

Reference standard:

Std#1 Amersham aged thorium nitrate

Std#2 Alfa Chemicals thorium oxide 99.9%

It was assumed that Th-230 would give the same response as Th-232 and Th-228 from these standards (i.e. cpm/pCi).

1 g sample

100 min count

CLV1 approximately 5.1 cpm

CLV2 approximately 0.5 cpm

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Radium-226

Analyst: Surinder Magon

Date analyzed: March 6, 1985

Sample decomposition: Potassium pyrosulphate fusion

In this procedure radium is isolated by first precipitating with lead sulfate and then selectively precipitating with barium sulfate. The radium in the precipitate is determined either by alpha spectroscopy or by gross alpha counting if thorium is known to be absent.

Reference standard:

Amersham RAY.44 Soln.#R1/300/54

Overall uncertainty ±3.7%

1 g sample

100 min count

CLV1 approximately 37.5 cpm

CLV2 approximately 2.6 cpm

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Lead-210

Analyst: Leonard Turple

Date analyzed: March 11, 1985

Sample decomposition: $HNO_3/HClO_4$ digestion

Extraction of Bi-210 with diethylammonium diethyldithiocarbamate (DDTC) from 2M HCl solution. After evaporation the organic extract is dissolved in nitric acid, diluted and BiOCl precipitated as the final counting form of Bi-210. The quantity of Bi-210 present is determined by beta counting with a low background counting system. The Pb-210 concentration is calculated from the equilibrium existing between it and the Bi-210.

Reference standard:

Amersham RBZ.44 soln.#S8/40/27

Overall uncertainty ±1.9%

5 g sample

100 min count

CLV1 approximately 45.9 cpm

CLV2 approximately 5.7 cpm

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Polonium-210

Analyst: Leonard Turple

Date analyzed: March 7, 1985

Sample decomposition: $HNO_3/HClO_4$ digestion

Polonium spontaneously plates onto silver or nickel from a 0.5 N HCl solution. The metal disk is counted by alpha spectroscopy or by gross alpha counting to determine the amount of Po-210. Standards are prepared in the same manner as the samples.

Reference standard:

Amersham RBZ.44 Soln.#S8/40/27

Overall uncertainty ±1.9%

5 g sample

100 min count

CLV1 approximately 37.7 cpm

CLV2 approximately 6.7 cpm